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| <p>Dr. Morikazu Nishiyama received his B.S. degree in Engineering from Osaka University in 1983, and his M.S. and Ph.D. degrees in Engineering from Osaka University in 1985 and 1988, respectively. He worked as a research assistant and then as an assistant professor in the Department of Engineering Science, Osaka University, from 1988 to 1993. He became an Associate Professor in the same department in 1993. His research interests are in the fields of membrane separation, porous silica, films, and electronic devices.</p>   |
| Research projects and selected papers  |
| <p>1. Ordered mesoporous silica membranes for<br/>         Ordered mesoporous silica membranes have been prepared by the sol-gel method. However, these membranes have a low permeability. It is difficult to prepare high-quality membranes. In this study, we have developed a new method for preparing high-quality ordered mesoporous silica membranes. The membranes prepared by this method have a high permeability and a low resistance. The permeability of the membranes is about 100 times higher than that of the membranes prepared by the sol-gel method. The resistance of the membranes is about 1/10 of that of the membranes prepared by the sol-gel method. The membranes prepared by this method are suitable for use in various applications.</p> |
| <p>2. Selective separation of organic vapors by<br/>         Organic vapors are separated by a selective membrane. The membrane is made of a porous material. The membrane has a high selectivity for organic vapors. The membrane is suitable for use in various applications.</p>  |

These materials are promising as a membrane and ultrafiltration.

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(2) Mesoporous silica with high thermal stability. H. Ueyama, Chem. Mater., 14, 146 (2002).

(3) Mesoporous silica with high thermal stability. H. Ueyama, Chem. Mater., 14, 146 (2002).

(4) Mesoporous silica thin films as a

membrane for ultrafiltration. H. Ueyama, Chem. Mater., 14, 146 (2002).

(5) Mesoporous silica thin films as a membrane for ultrafiltration. H. Ueyama, Chem. Mater., 14, 146 (2002).

(6) Mesoporous silica thin films as a membrane for ultrafiltration. H. Ueyama, Chem. Mater., 14, 146 (2002).

(7) Mesoporous silica thin films as a membrane for ultrafiltration. H. Ueyama, Chem. Mater., 14, 146 (2002).

(8) Mesoporous silica thin films as a membrane for ultrafiltration. H. Ueyama, Chem. Mater., 14, 146 (2002).

(9) Mesoporous silica thin films as a membrane for ultrafiltration. H. Ueyama, Chem. Mater., 14, 146 (2002).

(10) Mesoporous silica thin films as a membrane for ultrafiltration. H. Ueyama, Chem. Mater., 14, 146 (2002).



**Advances in Ceramic and Nano-Composite Membranes****Chair:**

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**Surface Engineering of Grafted Polymers on Inorganic Oxide Substrates for Membrane Pervaporation****Author Information:**

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**Abstract:**

Recent years have been marked by a growing interest in surface modification of pervaporation membranes by covalent end-attachment of polymer chains. The modification of inorganic membranes for pervaporation applications has been of special interest since the polymer chains alter the surface chemistry of the substrate (i.e., providing selectivity) while the mechanical strength of the membrane is retained. Covalently bonded polymers can be used in good solvent environments since dissolution of the polymer is prevented by its attachment to the substrate. In order to tune the performance of the

membrane one has to consider both the chemical and topological properties of the modifying polymer layer.

Past studies have been devoted to the macroscopic properties of the resulting hybrid organic/inorganic materials. However, interest is growing in the nanoscale features which result from the modification process. Since polymers are becoming an important tools in applications such as self-assembled monolayers and micromechanical devices, it is increasingly important to understand and quantify the characteristics of surface-bonded polymers so that surfaces can be engineered for specific applications. Poly(vinyl acetate) was chosen as a model polymer in order to analyze the surface properties resulting from surface modification by a two-step free-radical graft polymerization method. A variety of tools such as atomic force microscopy, thermogravimetric analysis, and scanning electron microscopy were used to examine the dependence of surface features on polymerization reaction conditions such as temperature and monomer concentration. Typical Flory radius of the polymers grafted in the study were found to be 110-170Å, while starting pore sizes of the inorganic membrane substrates used were 50-500Å. It was determined that polymer brush layer of 399Å was able to form on the membrane surface for the specific size and graft density (2.0-3.5 mg/m<sup>2</sup> surface) of the polymers produced by the present free radical graft polymerization.

Liquid separation membranes created using the above graft polymerization methods were found to efficiently separate organic mixtures of methanol and methyl-tert-butyl-ether with separation factors up to 100, and aqueous mixtures of TCE and water with separation factors of up to 370. Separation could be increased by increasing the polymer graft yield. Modified membranes displayed remarkably different behavior depending whether the polymer chain size was smaller or larger than the membrane pore size, suggesting that graft polymer size is as important a consideration as polymer chemistry in the modification of porous membrane materials.

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## Templating Strategies for Inorganic Molecular Sieve Silica Membranes

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#### Abstract:

Inorganic molecular sieve membranes (IMSM) are highly desirable for a variety of gas and vapor separations at elevated temperatures. Currently the most common IMSMs are amorphous carbon, polycrystalline zeolite and sol-gel silica membranes. Carbon membranes made by pyrolysis of polymeric precursors deposited on porous supports have potential for gas/vapor separations including CO<sub>2</sub>/CH<sub>4</sub> and H<sub>2</sub>/hydrocarbon separations.

On the other hand, polycrystalline zeolite membranes made by hydrothermal synthesis offer unique separation opportunities for a variety of permanent gas/isomer separations based on adsorption/diffusional differences of mixture components in zeolite pores. However, both classes of these IMSMs suffer from several problems such as poor processability, low permeation rates (due to thickness over 1 micron) and compromised selectivities due to susceptibility to cracking or undesirable intercrystalline porosity.

Sol-gel derived molecular sieve silica membranes on the other hand appear particularly attractive since they combine several advantages such as good processability, high permeation rates due to low thickness (e.g. below 100 nm) and tunable pore microstructure as regards pore size and chemical functionality. In this presentation we will demonstrate different templating strategies in order to obtain molecular sieve silica membranes with different pore sizes, useful for a variety of separations.

First, solvent templating (e.g. H<sub>2</sub>O or ethanol) is useful for creating pores in the range 3-4 Å, suitable

for a variety of permanent gas separations such as H<sub>2</sub>/N<sub>2</sub> or CO<sub>2</sub>/CH<sub>4</sub>. Second, molecular templating (e.g. TPABr) is useful for creating pores in the range 5-6 Å, useful for isomer separation such as n-butane/isobutane or p-xylene/o-xylene. We refer to these membranes as 'zeolite-like' membranes since they mimic the pore size of polycrystalline zeolite membranes (MFI) while obviating the disadvantages of their counterparts, e.g. large thicknesses or cracking and intercrystalline porosity. Finally, surfactant templating (e.g. CTAB, Brij56) is useful for creating ordered pores in the range 15-25 Å, leading to rapid formation of mesoporous silica membranes that can be used as intermediate support for the overlying solvent or molecular templated membranes described above. Microstructural characterization results (e.g. SEM, TEM, adsorption porosimetry) as well as permeation measurements with different permeant gases (H<sub>2</sub>, CO<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, SF<sub>6</sub>) or hydrocarbon isomers (n-butane, isobutane, p-xylene, o-xylene) will be presented in order to demonstrate the new separation opportunities offered by these novel sol-gel derived molecular sieve silica membranes.

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## Nanocomposite Membranes Derived from Zeolite 4A and Polyfurfuryl Alcohol

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### Abstract:

Nanocomposite Membranes Derived from Zeolite 4A and Polyfurfuryl Alcohol

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Previous studies have predicted that zeolite 4A is potentially a good candidate for air separation. As a result, the development of defect-free zeolite 4A membranes has attracted much attention. Several preparation techniques have been developed and these include conventional hydrothermal synthesis, microwave heating, dry-gel conversion, and secondary growth. However, the O<sub>2</sub>/N<sub>2</sub> selectivity so far has been limited to about 2.0 due to presence of inter-crystal defects.

Here we report a new strategy for fabrication of highly selective nanocomposite air separation membranes. We start with a hierarchical porous zeolite 4A film on a macroporous alumina tube by dip-coating of zeolite A nanoparticles. This film has well-defined bi-modal porosity at micro- and mesopore range [1-3]. Within this film, zeolite nanocrystals tightly contact one another, and thus zeolite channels remain well connected. The inter-particle mesoporosity can be eliminated by using a gas-impermeable phase as a filler. This strategy could be general for fabrication of other gas separation membranes.

In this presentation, we focus on polyfurfuryl alcohol (PFA) as inter-particle filler since crosslinked PFA has very low gas permeability [4] and can be easily deposited from its monomer – furfuryl alcohol through vapor phase polymerization [5].

SEM, elemental analysis, XRD and nitrogen adsorption-desorption measurements are used to characterize zeolite nanocrystals and nanocomposite membranes. Gas separation measurements show that the nanocomposite membranes have a good selectivity (e.g., O<sub>2</sub>/N<sub>2</sub> = 8).

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#### Modification of the Surface Characteristics of Anodic Alumina Membranes Using Sol-Gel Chemistry

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#### Abstract:

The objective of the work to be reported was to characterize the surface properties of anodic alumina membranes using streaming potential measurements and to modify those properties using sol-gel reactions to deposit titania or silica on the surface of the membranes. The motivation for this study arose from a desire to develop a better understanding of the relationship between the surface characteristics of an ultrafiltration membrane and its separation performance.

Streaming potential measurements involve the measurement of the voltage difference across a charged porous membrane resulting when electrolyte flows through the pores in the membrane. Streaming potential can be used to calculate the zeta potential, which is related to the charge of the membrane surface. By measuring the zeta potential in electrolytes of different pH, the isoelectric point of the membrane can be determined. The isoelectric point of unmodified anodic alumina membranes was found to be at  $\text{pH} \sim 8$ .

The surface properties of the alumina membranes were modified by depositing titania and silica on the pore surface within the membrane. The sol-gel reaction was carried out with hexane as solvent. With several exceptions, the only water provided for the reaction was that adsorbed to the alumina and that found in air. Modified membranes showed zeta potentials in neutral or basic electrolyte that were lower than those observed for unmodified membranes, indicating the isoelectric point to be at  $\text{pH} \sim 6.5$ . However, the zeta potential of these membranes was essentially the same as that observed for the unmodified membranes when measured with acidic electrolyte solutions. It was also observed that the zeta potential in neutral or basic electrolyte was the same as observed with the unmodified membranes after the modified membrane was exposed to an acidic solution. Similar observations were found when the sol-gel reaction was carried out in the presence of small amounts of added water or HCl.

A likely explanation for these observations is that the sol-gel reaction under the conditions used for these experiments results in the formation of nano-sized particles which are only weakly attached to the alumina surface. Exposure to an acidic environment results in the removal of these weakly bound particles, returning the membrane to its unmodified state. Experiments are currently underway whereby the sol-gel reaction is carried out in a basic environment. It is hoped that this change will result in the formation of a more networked metal oxide deposit that should be less susceptible to detachment in an acidic environment.

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Visualization of Three-Dimensional Pore Structure within Porous Films

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Abstract:

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## Sol-Gel Synthesis of Mesoporous Ceria and Zirconia Membranes-A Comparison Study

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#### Abstract:

CeO<sub>2</sub> and ZrO<sub>2</sub> find applications in various fields such as electrolytes, membranes and catalysis. Mesoporous CeO<sub>2</sub> and ZrO<sub>2</sub> films supported on porous ceramics can be used as membranes for filtration applications and as substrates for oxygen sensors and fuel cells. However, sol-gel preparation of these two oxide membranes, especially of CeO<sub>2</sub>, has been found difficult compared with to other oxides such as alumina. In this paper we will report sol-gel synthesis and properties of these two mesoporous ceramic membranes.

CeO<sub>2</sub> and ZrO<sub>2</sub> sols were used for coatings on alumina supports to reduce the pore size of the composite membranes. PVA (Polyvinyl alcohol) was used as a binder, which controls the porosity of the support without affecting the other microstructural properties. These coated membrane disks were dried under controlled humidity and temperature and then calcined. The pore size distribution of the sol gel derived unsupported membranes was determined by Adsorption Porosimetry. The phase structures of the supported membranes were identified by X-ray diffractometer and the average pore size of the composite membrane was determined by a home made gas permeation system.

XRD data show that the CeO<sub>2</sub> and ZrO<sub>2</sub> membranes have face centered cubic (FCC) structure and the average pore size of the membrane was found to be narrow than the support indicating that the membranes were pin-hole and crack free.

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#### Separation of Hydrogen using a Carbon Membrane

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#### Abstract:

The use of hydrogen as an alternative fuel supply promises to dramatically change many industries, especially the transportation industry. Every major vehicle manufacturer has under development alternative power cars based upon hydrogen powered fuel cells. Hydrogen has many characteristics that qualify it as a nearly perfect fuel for transportation. The most important is the potential to efficiently deliver energy without harmful emissions. The hydrogen fuel cell produces only water; however, emissions from the technology to produce the hydrogen must be carefully considered. Today, hydrogen is mostly produced from fossil fuels. Steam reforming of hydrocarbons such as natural gas to produce syngas, a mixture of hydrogen and carbon monoxide, is one example. Hydrogen can also be produced by partial oxidation of hydrocarbons, heating coal in the absence of oxygen, by partial burning of coal in the presence of steam, or by electrolysis. The reactions with coal make a mixture of H<sub>2</sub> with CO, CO<sub>2</sub> and other gases. The cheapest sources of new hydrogen are refinery fuel gas streams, PSA tail gas, FCCU gas, and hydrocracker/hydrotreater off-gas. These gas streams contain 30-80% hydrogen mixed with light hydrocarbons (C<sub>1</sub>-C<sub>5</sub>).

New membrane materials will be important for separation of hydrogen from each of the gases mentioned. Carbon molecular sieve membranes offer the advantage of achieving above Knudsen separation factors, operating at high temperatures and pressures, and may be less likely to foul or be affected by condensation of hydrocarbon vapors. High flux carbon membrane synthesis and results of small molecule separation from hydrogen above the Knudsen limit will be described.

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#### Amine-Modified Silica Membranes for Separation of Carbon Dioxide Under Ambient Conditions

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Abstract:

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Reducing CO<sub>2</sub> emissions for addressing climate change concerns is becoming increasingly important as the CO<sub>2</sub> concentration in the atmosphere has increased rapidly since the industrial revolution. Most of currently investigated mitigation processes require CO<sub>2</sub> in a concentrated form. However, CO<sub>2</sub> is emitted from large sources, such as coal-fired power plants, at ~15% concentration. Therefore, capturing CO<sub>2</sub> from dilute streams is an important step for many mitigation methods. Membrane separation methods are particularly promising due to potentially high CO<sub>2</sub> selectivities and fluxes. By a proper choice of the pore size and surface properties, the CO<sub>2</sub> transport across a membrane can be facilitated with respect to those of N<sub>2</sub> and O<sub>2</sub> leading to an efficient CO<sub>2</sub> separation process. However, current membranes suffer from a poor control over the pore size on the 2-4 nm scale and surface properties required for CO<sub>2</sub> separation.

We describe here novel mesoporous silica membranes with surface-attached amino groups promising for CO<sub>2</sub> separation from dilute streams. Defect-free 1-5 µm thick MCM-48 membranes displaying tunable 2-4 nm pores were fabricated on disk-shaped porous Al<sub>2</sub>O<sub>3</sub> and SS supports by solution growth and evaporation methods in the presence of cationic surfactants.

Several amino groups were attached to the membrane surface using silicon alkoxides, such as 3-aminopropyltriethoxysilane:  $(\text{Si-OH})_s + \text{C}_2\text{H}_5\text{-O-Si-R-NH}_2 \rightarrow (\text{Si-O-Si-R-NH}_2)_s + \text{C}_2\text{H}_5\text{OH}$ .


The amino group content and basicity critical for the CO<sub>2</sub> separation selectivity and flux were determined by TGA, elemental analysis, and an acid-base titration, respectively. CO<sub>2</sub>-N<sub>2</sub> separation on amine-modified silica membranes was investigated at 20-70°C and  $p = 0.2-1$  atm as a function of the pore size (2-4 nm) and the nature of the amino group using a Wicke-Kalenbach permeability cell. The membrane performance was evaluated with respect to permeance and separation selectivity, determined by a GC analysis of the permeate.

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## Development of porous silicon-based miniature fuel cells

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**Abstract.** Nowadays the rise in portable electronics requires energy sources compatible with the environmental constraints. We demonstrate, in this paper, how microfabrication techniques allow the development of low-cost miniature fuel cells fully integrated on silicon. Contrary to usual proton-conducting membranes made of ionomers ensuring the proton conductivity of proton-exchange membrane fuel cells (PEMFCs), we present here another way to proceed. It consists in the chemical grafting of molecules bearing acid groups on the pore walls of a porous silicon membrane to mimic the structure of an ionomer, such as Nafion®. We obtain an inorganic, dimensionally stable, proton-conducting membrane with many optimizable parameters such as the pore size and the pore structure of the membrane or the nature of the grafted molecules. Moreover, the use of a silicon substrate offers advantages of serial and parallel integration, the possibility of encapsulation by wafer bonding and gas feed and electrical contacts may be included into the membrane etching process, thanks to simple KOH wet etching processes and metal sputtering.

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